भारतीय मानक
Indian Standard

IS 3025 (Part 57) : 2021

जल एवं अपशिष्ट जल के नमूने लेने तथा परीक्षण (भौतिक एवं रासायनिक) की पद्धतियाँ

भाग 57 बोरोन

(दूसरा पुनरीक्षण)

Methods of Sampling and Test (Physical and Chemical) for Water and Wastewater

Part 57 Boron

(Second Revision)

ICS 13.060.50

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Water Quality Sectional Committee, CHD 36

FOREWORD

‘This Indian Standard (Part 57) (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Water Quality Sectional Committee had been approved by the Chemical Division Council’.

Boron is not found free in nature but is always bound to oxygen. It occurs generally as ortho-boric acid and as alkali metal and alkaline earth metal berates. Boron constitutes only about 3 ppm of the earth’s crust. The natural borate content of groundwater and surface water is usually small. The borate content of surface water can be significantly increased as a result of wastewater discharges, because borate compounds are ingredient of domestic working agents. Naturally occurring boron is present in ground water primarily as a result of leaking from rocks and soils containing berates and boro-silicates. It is mainly found in soils from 18 ppm to 63 ppm. The majority of Earth’s boron occurs in the oceans, with an average concentration of 4.5 mg/1. The amount of boron in fresh water depends on geochemical nature of the drainage area, proximity to marine coastal region and inputs from industrial and municipal effluents. Boron is an essential element for plant growth as micro nutrient but if present in amount higher than 2 ppm, it is deleterious to some plants.

The technical committee responsible for formulation of IS 3025 : 1964 ‘Methods of sampling and test (physical and chemical) for water used in industry’ decided to revise the standard and publish it in separate parts. This standard superseded **29** of IS 3025 : 1964 and was one among the different parts being published under IS 3025 series of standards. The first revision of this standard was published in 2005 which prescribed five methods for detection of boron in water and wastewater.

In this second revision the following changes have been made:

a) Curcumin method has been modified ;
b) Carmine method has been added;
c) ICP method has been updated after incorporating ICP-MS, ICP-OES and ICP-AES; and
d) Titration method, Flow injection method, and ion chromatographic method have been deleted.

In the preparation of this standard, considerable assistance has been derived from the method No. 4500-B of — Standard methods for the examination of water and wastewater, published by the American Public Health Association, Washington, USA, 23rd Edition, 2017.

The composition of the committee, responsible for the formulation of this standard is given at Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTE WATER

PART 57 BORON

(Second Revision)

1 SCOPE

This standard prescribes three different methods for the determination of boron:

a) Curcumin method from 0.10 mg/l to 1.0 mg/l;
b) Carmine method from 1.0 mg/l to 10 mg/l; and
c) Induced coupled plasma method.

2 REFERENCES

The Indian Standards listed below contain provisions which, through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
264 : 2005	Nitric acid (*third revision*)
3025 (Part 2) : 2019	Methods of sampling and test (physical and chemical) for water and wastewater: Part 2 Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)
3025 (Part 65) : 2014	Methods of sampling and test (physical and chemical) for water and wastewater: Part 65 Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of 62 elements
7022 (Part 1) : 1973	Glossary of terms relating to water, sewage and industrial effluents: Part 1
7022 (Part 2) : 1979	Glossary of terms relating to water, sewage and industrial effluents: Part 2

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 7022 (Part 1) and 7022 (Part 2) shall apply.

4 SAMPLE PRESERVATION

The sample bottles should be cleaned thoroughly with 6N nitric acid prior to final rinsing with deionized water. The water samples should be collected and stored in polythene bottles or alkali resistant, boron – free glassware and silica containers. For preservation, the sample should be acidified with HCl (2 ml AR grade concentrated HCl in 1 l of sample to bring down the *p*H below 2).

5 CURUMIN METHOD

5.1 Principle

The sample of water containing boron when acidified will react with a curcumin reagent and on evaporation it will produce a red coloured product known as rosacyaninine. This product is extracted in a suitable solvent and the red color is compared with standards visually or photo metrically.

5.2 Interferences

The high quantity of hardness will cause interference due to its insolubility in ethyl alcohol and is to be removed by passing the original sample through a column of strongly acidic Cation-Exchange resins. The concentration of nitrates above 20 mg/l causes interference. Phosphate does not interfere. The measurable range is from 0.10 mg/l to 1.0 mg/l.

5.3 Apparatus

5.3.1 *Spectrophotometer,* for use at 540 nm, light path 1 cm.

5.3.2 *Evaporating Dishes,* 100 to 150 ml capacity of high silica glass, platinum crucible or any other suitable material.

5.3.3 *Water Bath*, to be set up at (55 ± 2) °C.

5.3.4 *Glass Stoppered Volumetric Flasks*, 25 ml and 50 ml capacity.

5.3.5 *Ion Exchange Column,* 50 cm long and 1.3 cm in diameter.

5.4 Reagents

5.4.1 *Stock Boron Solution*

Dissolve 571.6 mg of anhydrous boric acid (H_3BO_3) in distilled water and dilute it to 1 000.00 ml (1 ml = 100 µg B). The boric acid loses weight when dried at 105 °C and hence ACS grade reagent may be used with the bottle always stoppered to prevent entry of moisture.

5.4.2 *Standard Boron Solution*

Dilute 10.0 ml stock boron solution to 1 000.0 ml. (1 ml = 1.00 µg B).

5.4.3 *Curcumin Reagent*

Dissolve 40 mg of finely ground curucumin (Eastman no 1179 or equivalent) and further add 5.0 gm of oxalic acid to 80 ml of 95 percent ethyl alcohol. Then add 4.20 ml concentrated hydrochloric acid and makeup the volume upto 100.00 ml with ethyl alcohol in a 100.00 ml volumetric flask. Filter the reagent in case it is turbid. 95 percent isopropyl alcohol can also be used in place of ethyl alcohol. This reagent can be stored in a refrigerator for several days.

5.4.4 For the removal of interferences, strongly acidic cation exchange resin can be used.

5.4.5 *Hydrochloric Acid (HCl), 1 + 5*

5.5 Procedure

5.5.1 *Preparation of Calibration*

Pipet out 0 (blank), 0.25, 0.50, 0.75 and 1.0 µg boron in the evaporating dishes of similar size and type. Further add distilled water in each standard and then make up the volume upto 1.0 ml. Add 4.0 ml of curucumin reagent to each of them and gently swirl it to mix the contents. Then set the water bath at (55 ± 2) °C for 80 min and float the dishes in a water bath that is sufficient for complete drying and removal of HCl. As soon as it cools to room temperature, add 10 ml of 95 percent ethyl alcohol to each dish and gently stir it by using a polyethylene rod and insuring dissolution of red coloured product.

Wash the contents to a 25 ml volumetric flask using 95 percent ethyl alcohol and make up the volume upto the mark with 95 percent ethyl alcohol. Read the absorbance of standards and blank at 540 nm and prepare a calibration curve from 0 µg to 1µg of boron.

5.5.2 *Sample Treatment*

Take 1 ml of sample in case the boron is within the range else make appropriate dilution such that 1 ml of it contains approximately 0.5 µg boron and follow the similar procedure as adopted for standards.

5.5.3 *Removal of High Hardness and Cation Interferences*

Prepare an ion exchange column of dimension 20 cm * 1.3 cm diameter and charge it with strongly acidic cation exchange resin. Further, back wash the column by using distilled water in order to remove air bubbles. Cover the resin with the liquid at all times. Now pass 50 ml of 1+ 5 HCL through the column at a rate of 0.2 ml of acid per ml of resin in the column per min. Now wash the column free of acid using distilled water.

Pipette out 25 ml or even a smaller sample and then dilute it to 25 ml and further pass it through the column prepared with resin. Collect the clear water and then proceed for the development of color as per the method given above.

5.5.4 *Calculation*

$$\text{Boron, mg/l} = \frac{A_2 \times C}{A_1 \times S}$$

where

A_2 = absorbance of sample,
C = mass of boron of standard taken, in µg,
A_1 = absorbance of the standard, and
S = volume of sample, in ml.

6 CARMINE METHOD

6.1 Principle

The sample of water containing boron will react with a bright red colored carmine reagent/ carminic acid in concentrated sulphuric acid media, to discharge a bluish red or blue color depending on the concentration of boron. The measurable range is from 1.0 mg/l to 10.00 mg/l

6.2 Apparatus

6.2.1 *Spectrophotometer*, for use at 585 nm, light path 1 cm.

6.2.2 *Test Tubes or Flasks*, 30 ml capacity.

6.3 Reagents

6.3.1 *Stock Boron Solution*

Take 571.6 mg of anhydrous boric acid and dissolve it in distilled water. Further dilute it to 1 000.00 ml

(1ml = 100 µg B). During the process of drying, boric acid may lose weight and hence ACS grade reagent maybe used with bottle that is stoppered at all times to prevent entry of moisture.

6.3.2 *Standard Boron Solution*

Dilute about 10.00 ml of stock boron solution to 1 000.00 ml. (1ml = 1.00 µg B).

6.3.3 *Carmine Reagent*

Dissolve 920.00 mg of carmine N.F 40 or carminic acid, in a 1 l of conc sulphuric acid (If zero absorbance is not achieved dilute carmine 1 + 1 with concentrated sulphuric acid to replace the above reagent).

6.3.4 *Hydrochloric Acid, HCl (1+11)*

6.3.5 *Sulphuric Acid, concentrated*

6.4 Procedure

6.4.1 *Low Level Sample Concentration*

If the sample has less than 1 mg B/l then pipette out a small amount of sample that contains at least 20 µg B in a platinum dish and make it alkaline by using 1N sodium hydroxide. Further, evaporate it to dryness by keeping it on a hot water bath. Destroy any kind of organic matter present in it by heating at 550 °C. Acidify the cooled residue by using 2.5 ml (1 + 11) Hydrochloric acid and triturate with a rubber police man to dissolve. Centrifuge it and take out a clear 2.00 ml sample into a 30 ml of test tube and treat reagent identically.

6.4.2 *Color Development*

Pipette out series of boron standards solution along with (blank, 100.00 µg, 250.00 µg, 500.00 µg, 750.00 µg and 1000.00 µg) in 100 ml with distilled water. Again pipette 2.00 ml of each standard solution in a smaller flask or else in a 30 ml test tube.

Treat the blank, calibration standards and the sample in a similar way. Then add 2 drops of concentrated hydrochloric acid and carefully introduce 10 ml of concentrated sulphuric acid. Mix it well and let it cool to the room temperature. Further add 10.0 ml of carmine reagent, again mix it well and after a period of 45 min to 60 min measure the absorbance at 585 nm in a cell of 1cm or longer light paths with blank as reference.

Presence of air bubbles should be thoroughly checked in the optical cell while readings are taken. Bubble may result due to the incomplete mixing of reagents.

6.5 Calculation

$$\text{Boron, mg/l} = \frac{M}{V} \times D$$

where

M = mass of boron in µg,

V = volume of sample in ml, and

D = Dilution correction (D = 1 for undiluted samples and 1.25 if concentrated for low level sample) (*see* **6.6.1**).

7 INDUCTIVELY COUPLED PLASMA METHOD

7.1 Inductively Coupled Plasma Atomic Emission Spectrometric (ICPAES) Method

7.1.1 *Principle*

The ICPAES technique is based on the excitation of atoms by an argon plasma generated by the application of high frequency RF current. The sample is introduced into the argon plasma through a nebulizer. The emission spectra of the atom is measured using a suitable grating and photomultiplier system. The ICPAES technique is suitable for the analysis of boron in different types of water samples like natural water, potable water, domestic and industrial waste waters. Detect ion limits of about 5 µg/l can be achieved using the wavelength of 249.77 nm and upper detection limit of about 50 µg/l can be achieved using the wavelength of 249.66 nm.

7.1.2 *Apparatus*

Inductively coupled Plasma Atomic Emission Spectrometer (ICPAES). Either sequential or simultaneous (polychromatic system) maybe employed. Instrument setting as recommended by the manufacturer shall be followed.

7.1.3 *Reagents*

7.1.3.1 *Nitric acid concentrated* — Conforming to IS 264.

7.1.3.2 *Stock boron solution A* (*1 000* µg/*ml*) — Dissolve 571.6 mg of anhydrous boric acid (H_3BO_3) in distilled water and dilute to 100 ml(1ml = 1000 µg B/ml). Boric acid may lose weight on drying hence ACS grade reagent maybe used with bottle always stoppered to prevent entry of moisture.

7.1.3.3 *Standard boron solution B* (*100* µg/*ml*) — Dilute 10 ml stock boron solution A make up to 100 ml with deionized water.

7.1.3.4 *Standard boron solution C (10* µg*/ml)* — Dilute 10 ml of solution B to 100 ml with deionized water.

7.1.4 *Procedure*

7.1.4.1 Follow the procedures as described in manufacturer's instrument operating manual.

7.1.4.2 *Standardization*

Standardize the instrument and obtain a wavelength profile using a solution of 10 µg/ml of boron solution (*see* **7.3.4**).Using the 10 µg/ml boron solution (*see* **7.3.4**) and demineralized water as high and low standards obtain a calibration curve. Check the reproducibility of the data by repeatedly aspirating a solution of an intermediate concentration level (5 µg/ml). Choose a wavelength of 249.773 nm for the analysis at low concentrations of boron and 249.66 nm for high concentrations. Prepare a test standard of 1 ppm of boron by diluting 10 ml of solution C to 100 ml with deionized water. Use this standard for calculation of precision over short and long periods. Use freshly prepared solutions for all the experiments.

7.1.4.3 *Analysis of sample*

Filter the samples solution through a 0.45 micron filter membrane or medium textured filter paper to avoid clogging or choking of the nebulizer. However, in the case of water samples containing high salt content (>10 mg/ml), separate the matrix and inject the sample into the plasma for analysis.

7.1.5 *Calculation*

Determine the peak height or peak area of the sample and read the boron concentration from the standard calibration curve.

7.1.6 *Precision and Accuracy*

The precision of ICPAES measurement is about 2-3 relative standard deviation (RSD).

7.2 Other Induced Coupled Plasma Method

Boron can also be determined by induced coupled plasma optical emission spectroscopy with reference to procedure given in IS 3025 (Part 2). Likewise, induced coupled plasma mass spectroscopy with reference to procedure given in IS 3025 (Part 65) can also be used for the determination of boron.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Water Quality Sectional Committee, CHD 36

Organization	*Representative(s)*
Chief Scientist, EPTRI, Hyderabad	Shri N. Raveendhar (***Chairman***)
Avantha Centre for Industrial Research and Development, Yamuna Nagar	Dr Nishi Kant Bhardwaj Dr Sunil Kumar (*Alternate*)
Bhabha Atomic Research Centre, Mumbai	Dr S. K. Sahu Shri I. V. Saradhi (*Alternate*)
Central Ground Water Board, Faridabad	Shri G. C. Pati Shrimati Rumi Mukherjee (*Alternate*)
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Central Pollution Control Board, New Delhi	Shri N. C. Durgapal Dr J. C. Babu (*Alternate*)
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Gujarat Pollution Control Board, Gandhinagar	Dr D. N. Vansadia Shri K. B. Vaghela (*Alternate*)
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Indian Institute of Toxicology Research, Lucknow	Dr S. C. Barman Dr Satyakam Patnaik (*Alternate*)
Indian Water Works Association	Shri Vijay Charhate

Organization	*Representative(s)*
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Member Secretary

Ms Shubhanjali Umrao
Scientist 'B' (CHD), BIS

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Amendments Issued Since Publication

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